J Mater Sci (2011) 46:2627–2634
DOI 10.1007/s10853-010-5117-9

A Study on flexural properties of wildcane grass fiber-reinforced polyester composites

A. V. Ratna Prasad · K. Mohana Rao · A. V. S. S. K. S. Gupta · B. V. Reddy

Received: 17 July 2010 / Accepted: 25 November 2010 / Published online: 14 December 2010

Abstract The main objective of this study is to introduce a new natural fiber as reinforcement in polymers for making composites. Wildcane grass stalk fibers were extracted from its stem using retting and chemical (NaOH) extraction processes. These fibers were treated with $KMnO_4$ solution to improve adhesion with matrix. The resulting fibers were intentionally reinforced in a polyester matrix unidirectionally, and the flexural properties of the composite were determined. The fibers extracted by retting process have a tensile strength of 159 MPa, modulus of 11.84 GPa, and an effective density of 0.844 g/cm^3. The composites were formulated up to a maximum fiber volume fraction of 0.39, resulting in a flexural strength of 99.17 MPa and flexural modulus of 3.96 GPa for wildcane grass fibers extracted by retting. The flexural strength and the modulus of chemically extracted wildcane grass fiber composites have increased by approximately, 7 and 17%, respectively compared to those of composites made from fibers extracted by retting process. The flexural strength and the modulus of $KMnO_4$-treated fiber composites have increased by 12 and 76% over those of composites made from fibers extracted by retting process and decreased by 3 and 48% over those of composites made from fibers extracted by chemical process, respectively. The results of this study indicate that wildcane grass fibers have potential as reinforcing fillers in plastics in order to produce inexpensive materials with high toughness.

A. V. R. Prasad (✉) · K. M. Rao
Department of Mechanical Engineering, V R Siddhartha Engineering College, Vijayawada 520 007, India
e-mail: rp_atluri@yahoo.co.in

A. V. S. S. K. S. Gupta
Department of Mechanical Engineering, JNTU College of Engineering, Hyderabad 500 072, India

B. V. Reddy
Faculty of Engineering and Applied Science, University of Ontario Institute of Technology, Oshawa, Canada

Introduction

The use of natural vegetable fibers as reinforcements in polymer composites to replace synthetic fibers like glass and carbon is currently receiving increasing attention because of the advantages, including cost effectiveness, low density, high specific strength, bio-degradability as well as their availability as renewable resources. Natural fibers are classified according to their source; plants, animals, or minerals. In general, it is the plant fibers that are used to reinforce plastics in the composite industry. Many varieties of plant fibers exist, such as bamboo, jute, hemp, and flax extracted from stem of the plant, sisal, and abaca from leaf and coir and cotton from fruit of the plant. The abundance of these raw materials is also a concern to the manufacturing industry, and the responsibility to use evermore greener technologies has made this area of research more interesting worldwide.

Owing to the poor compatibility toward polymers resulting from the hydrophilicity of these plant fibers, many studies have been carried out to improve the interfacial bonding, either by surface modification or by plasticization of the fibers [1–3]. The chemical composition, properties of sisal fibers, and their composites by incorporating untreated and treated fiber in different matrices were reported [4, 5]. It was observed that the mechanical process yields good quality of fiber compared to retting process, even though the yield is more in the later case. The mechanical properties of sisal, banana, and hemp fiber composites using novolac resin, with and without maleic anhydride treatment were studied [6]. The properties like Young's modulus, flexural modulus, and impact strength increase with maleic anhydride treatment.

The physical and mechanical properties of banana fiber strand-reinforced polyester composites were reported [7]. A considerable increase in flexural strength, modulus, and fracture toughness was observed for the composite with 30% fiber by weight. The experiments of tensile and flexural tests were also carried out using woven banana fiber-reinforced epoxy composites [8]. It was found that the maximum tensile strength in x and y direction is 14.14 and 3.398 MPa, respectively and for Young's modulus the values are 976 and 863 MPa in x and y directions, respectively. The maximum flexural strength and modulus in x direction was recorded as 26.181 MPa and 2.685 GPa, respectively.

The tensile and flexural properties of bamboo/polyester and bamboo/epoxy composites were reported [9]. Bamboo fiber-reinforced unsaturated polyester composite has marginally lower properties than bamboo fiber-reinforced epoxy composite. The effects of phenol formaldehyde as bonding agent and Si69 as coupling agent on curing characteristics and mechanical properties of bamboo fiber filled natural rubber composites were also studied [10, 11]. The presence of bonding agent as well as the coupling agent enhanced the mechanical properties like tensile strength, modulus, tear strength, and elongation at break. Some studies were also carried out on the flexural behavior of bamboo fiber-reinforced mortar laminates [12]. It was reported that the flexural strength values of bamboo fiber-reinforced mortar laminates have been improved more than 90 MPa. Recently, studies on PP-based composites using steam exploded bamboo fibers have increased the tensile strength and modulus by about 15 and 30%, respectively [13]. Bamboo fibers extracted by different methods were mixed in poly lactic acid (PLA) and the mechanical properties like impact strength and heat resistance were determined [14]. The impact strength of PLA/MFB (medium bamboo fiber bundles) composite significantly increased and the addition of BF improves thermal properties and heat resistance of PLA/BF composites because of the constraint of deformation of PLA in conjunction with crystallinity promoted by anneal at 110 °C.

The effect of fiber surface wettability, alkali treatment, and different aging conditions on the physical, mechanical, and thermal properties of longitudinally oriented jute fiber-reinforced polyester composites were investigated [15–17]. It was observed that the flexural modulus and elongation at break were improved by using NaOH treatment. The manufacture and mechanical properties of flax fiber-reinforced epoxy composites with different fiber treatments were reported [18]. The results indicated that the strength and modulus values are considerably improved. Composites of an aliphatic polyester (Bionolle) with natural flax fibers are prepared by batch mixing and the effect of processing conditions on fiber length distribution, and the dependence of the composite mechanical properties on fiber content were investigated [19]. A 30% increase in strength is observed when natural fibers (25 vol%) are substituted by fibers containing acetate groups. The compressive strength of unidirectional flax fiber-epoxy composites was studied [20]. It was observed that the increase in compressive strength is proportional to the amount of melamine formaldehyde (MF) resin in the composite, and the presence of MF resin in the fibers lowers their tensile strength and subsequently the tensile strength of the resulting composite.

The influence of surface modifications of coir fibers involving alkali treatment, bleaching, and vinyl grafting on the performance of coir-polyester composites was studied [21]. The mechanical properties of composites like tensile, flexural, and impact strengths increased as a result of surface modifications. Coir-polyester composites were prepared up to 80% fiber weight fraction with two molding pressures [22]. It was concluded that random oriented coir fiber composites are having low flexural strength compared to that of bare polyester. Coir/rubber composite with the fiber volume fraction of 60% was fabricated by using the heat press technique [23]. It was observed that the temperature variation (130–160 °C) during the heat press process has no significance on the tensile strength of the composites. The effects of aging on the tensile properties and dimensional stability of pine apple leaf fiber-polyester composites were studied [24]. Aging studies showed a decrease in tensile strength of the composites.

Composites of polypropylene filled with 30% wheat straw fibers extracted by both mechanical and chemical processes were prepared, and their mechanical properties were also evaluated [25]. These composites exhibited significantly enhanced properties compared to virgin polypropylene. The mechanical properties of long and random hemp and kenaf fiber-reinforced polyester composites with the fibers as-received condition and also alkalized with 6% NaOH solution were reported [26]. It was observed that alkalized fiber composites gave higher flexural strength and modulus compared with composites made from as received fibers. Composites of different kinds of natural fibers like cotton, hemp, and kenaf were processed with a fiber mass proportion of 40% and PLA by compression molding [27]. The characteristics like tensile strength, %elongation, tensile modulus, and impact strength varied markedly depending on the characteristics of raw fibers and fiber bundles.

The use of short palm tree lignocellulosic fibers as a reinforcing phase in polyester and epoxy matrices has been reported [28]. It was shown that the interfacial adhesion was improved with the esterification of fiber in alkaline medium using acetic and maleic anhydrides, and succeeded to improve the mechanical properties of epoxy-based composites only. The behavior of untreated and chemically pre-treated peach palm fibers as reinforcement in polyester composites was studied [29]. The results of tensile strength show that the composites with treated fibers do not have significantly different properties to those with untreated fibers and the impact results show the treatment with H_2O_2 allowed an increase in the impact resistance of the composites at a fiber fraction of 10 wt%. Woven betel palm composites were prepared in both untreated and treated forms with different fiber volumes up to 9% [30]. The flexural properties and impact strength of woven betel palm composites improve with the addition of fiber up to 7 vol% and the drop in flexural properties was observed in 9 vol% of fiber. Tensile and impact properties of fully green composites reinforced with mercerized ramie fibers were studied [31]. Results of tensile tests showed that unidirectional composites using mercerized ramie yarns exhibited two to three times larger fracture strain without a marked decrease in strength than composites using untreated yarns. Fibers were also extracted from zostera marina sea grass collected from the baltic coast [32]. It was observed that single fiber is having stiffness values up to 28 GPa.

The mechanical and dielectric properties of vakka fiber-polyester composites have been studied and compared with sisal, bamboo, and banana composites [33]. It was reported that the dielectric strength of vakka fiber composites increases with increase in volume fraction of fiber unlike other composites in the study. Recently, a review on bast fibers like flax, hemp, jute, and kenaf and their composites were reported to examine the growth, harvesting, and fiber separation techniques suitable to yield fiber of appropriate quality [34]. Table 1 lists some of these research papers concerning the mechanical properties of few natural fiber-reinforced polymer composites for comparison with the results of this study.

In the past, several studies have been carried out on various natural fibers extracted from stem, leaf, or fruit and their composites, but the study of wildcane grass fiber is still scarce. And the fact that wildcane grass fiber obtained directly from the stem of a plant of a natural resource and its renewability make it more attractive in terms of sustainability and environmental awareness. Wildcane grass (Scientific name: *Saccharum spontaneum*) grows all over Asia and also known as false sugar cane or khagra grass (Fig. 1). It belongs to a family of gramini and grows up to 3 m in height where soil is rich in moist locations along lake and river beds. Wildcane grass grows naturally without using any agrochemicals. It has 2–3 m stiff slender culms with 0.6–1.25 cm in diameter and it finds applications to prevent erosion of sand in river banks, thatching of huts, and also as animal feed. It is also believed that the huts thatched by this grass are durable for a long period of time because of its inherent strength and stiffness.

The overall objective of this work is to investigate the fiber extraction by different methods and its treatment from wildcane grass and the use of these fibers as reinforcement in polymer matrix. A combination of retting, chemical, and manual methods is used to extract the fibers. These fibers are reinforced into unsaturated polyester resin to make composites at various volume fractions of fiber and tested to evaluate their flexural properties.

Methods and materials

The unsaturated polyester resin of the grade ECMALON 4411 was purchased from Ecmass Resins (Pvt) Ltd., Hyderabad, India. The resin has 1.258 g/cm^3 density, 500 cps Viscosity at 25 °C, and 35% monomer content. Cobalt naphthanate as accelerator and methyl ethyl ketone peroxide (MEKP) as catalyst were used.

Extraction of fibers

The extraction of fibers was done by two methods namely (a) Retting and (b) Chemical methods, with both followed by manual extraction. In the first method, after cutting the wildcane grass at their base, the leaves at the nodes and end of the culms were trimmed. After trimming, the culms were dried in shade for a period of 1 week. The node portions were removed by cutting and the culms were separated into pieces of 100 mm length. These cylindrical pieces which contain lignin in the central portion are made into strips by peeling them in longitudinal direction and the lignin is removed from them. These strips were kept in water for a period of about 3–5 days in order to soften them, and then subjected to a mechanical process by beating them gently with a plastic mallet in order to loosen and separate the fiber. The resulting fiber bundle is scraped and combed until individual fibers are obtained. This fiber was denoted by wildcane grass (M).

In the second method, the strips were soaked in 0.1 N NaOH solution (4 g of NaOH crystals per 1 L of water) for different periods. After a series of experiments, a period of 72 h is taken as optimum period for chemical treatment. After this treatment, the strips were washed in water and were subjected to the above mechanical process for separation of fibers. This fiber was denoted by wildcane grass (C).

Surface treatment of fibers

The extracted wildcane grass fibers (M) and (C) of 30 g each were taken separately and are soaked with 11 parts of

Table 1 Comparison of mechanical properties of various natural fiber-reinforced composites from literature

Composite	Fiber type	Treatment	Form	Fiber content	Property	Value before treatment (MPa)	Value after treatment (MPa)	% increase/ decrease in property	Cited reference
Sisal-polyester	Leaf	Silane	Unidirectional	50% vol	T. S	29.66	34.14	15	[4]
					Y. M.	1150	1750	52	
					E. B. %	9.52	5.71	40	
					F. S.	59.57	96.88	63	
					F. M.	11940	19420	63	
		N-substituted methacrylamide			T. S.		39.48	33	
					Y. M.		2060	79	
					E. B. %		9.75	2.4	
					F. S.		76.75	29	
					F. M.		15350	29	
Pine apple-polyester	Leaf	–	Random	40% wt	T. S.	63.3	–	76	[24]
					Y. M.	2519		432	
					E. B. %	5.0		312	
Jute-polyester	Stem	10% NaOH	Rovings	30% vol	T. S.	68.7	61.8	10[b]	[15]
					Y. M.	1400	1220	12.85[b]	
					E. B. %	6.15	6.6	7.3	
					F. S.	115	96	16.5[b]	
					F. M.	4250	4310	1.4	
					I. S. J/m	84.2	80	5[b]	
Banana- polyester[a]	Stem	–	Strands	30% wt	F. S.	97	–	32	[7]
					F. M.	6500		58.5	
Coir-polyester	Seed	5% NaOH	Non woven mat	17% wt	F. S.	52	60.4	17	[21]
					T. S.	22	27	22.7	
					I. S. J/m	433.5	634.6	46	
Flax-Epoxy	Stem	PVA	Unidirectional mat	40% vol	T. S.	105	156	48.5	[18]
					Y. M.	9500	10160	7.0	
		Urea		50% vol	T. S.	118.5	121.5	2.5	
					Y. M.	11860	13960	17.7	
Bamboo-polyester[a]	Stem	–	Mat	68% vol	F. S.	106.9	–	–	[9]
					F. M.	7520			
Betel palm-polyester	Leaf	6% NaOH	Woven	7% vol	F. S.	42	63	50	[30]
					F. M.	2800	3150	12.5	
					I. S. kJ/m^2	7.75	8.0	3.2	
Peach palm-polyester	Leaf	5% NaOH	Unidirectional mat	10% wt	T. S.	10.21	11.38	11.5	[29]
					Y. M.	214.49	260.35	21.4	
					E. B. %	1.53	2.01	31.3	
Hemp-polyester	Stem	6% NaOH	Unidirectional	60% vol	F. S.	75	100	33.4	[26]
					F. M.	7500	9500	26.7	
					I. S. kJ/m^2	75	50	33.4[b]	
Kenaf-polyester	Stem	6% NaOH	Unidirectional	64% vol	F. S.	30	122	406	
					F. M.	3500	13000	371	
					I. S. kJ/m^2	62	72	16	
Vakka-polyester[a]	Leaf	–	Unidirectional	37% vol	T. S.	66	–	440	[33]
					Y. M.	1790		406	
				39% vol	F. S.	93.79		42	
					F. M.	3320		128	
Wildcane grass-polyester	Stem	$KMnO_4$	Unidirectional	39% vol	F. S.	99.17	111	12	This study
					F. M.	3960	7000	76	

T. S. Tensile strength, *Y. M.* Young's modulus, *E. B* Elongation at break, *F. S.* Flexural strength, *F. M.* Flexural modulus, *I. S.* Impact strength

[a] Reults for untreated fiber composites and % increase over pure matrix

[b] % decrease

Fig. 1 Photograph of the wildcane grass plant

$KMnO_4$ with a concentration of 0.055% solution in acetone medium and the fibers were air dried. After surface treatment, the wildcane grass (M) and wildcane grass (C) fibers were denoted by wildcane grass (MK) and the wildcane grass (CK), respectively.

Composite preparation

Hand-lay-up method was adopted to fill up the prepared mold with an appropriate amount of polyester resin mixture and layers of unidirectional wildcane grass fibers, such that starting and ending with layers of resin. The quantity of accelerator and catalyst added to resin at room temperature for curing was 1.5% by volume of resin each. Fiber deformation and movement should be minimized to yield good quality, unidirectional fiber composites. Therefore, at the time of curing, a compression pressure of 0.05 MPa was applied using dead weights on the mold and the composite specimens were cured for 24 h. The specimens were also post-cured at 70 °C for 2 h after removing from the mold. Fiber configuration and volume fraction are the two most important factors that affect the properties of the composite. In this study, configuration is limited to unidirectional, continuous wildcane grass fibers equal to the length of specimen i.e., 100 mm and the composite samples were prepared with five different volume fractions of wildcane grass fibers.

Testing of fibers and composites

A 2 ton capacity electronic tensometer model METM 2000 ER-I supplied by M/s Microtech, Pune, India, was used for testing of samples. The tensile strength of various wildcane grass fibers was measured in accordance with ASTM D3379-75. Each fiber specimen was prepared by mounting on a stiff cardboard piece with a gauge length of 50 mm. The ends of the fibers were glued on to the cardboard with epoxy resin and tested at a cross head speed of 0.5 mm/min. Tensile strength and tensile modulus were calculated from the load-elongation data and cross-sectional area of fibers. The diameter of the fibers was measured using an optical microscope at five different locations along the gauge length and the average cross-sectional area was calculated by assuming that the fibers were cylindrical in shape. Since these natural fibers are highly irregular in shape, the test was conducted for ten specimens to get a valid average. However, the variation in properties of these fibers depends on its source, age, etc.

Three point bend tests were performed in accordance with ASTM D790 M test method I, procedure A to measure flexural properties. The samples were 100 mm long by 25 mm wide by 3 mm thick and five identical specimens were tested for each composition. In three point bend test, the outer rollers are 64 mm apart and the samples were tested at a cross head speed of 0.5 mm/min. A three point bend test is chosen because it requires less material for each test and eliminates the need to accurately determine center point deflections with test equipment. The tests were conducted at 25 °C and 50% relative humidity in the laboratory atmosphere. The flexural modulus and the maximum composite stress were calculated using the relationships given in the study [35]. The density of fiber and its composites were measured using pycnometric procedure.

Results and discussion

The density and tensile properties of various natural fibers along with wildcane grass fibers were summarized in Table 2 [36–38]. The density of wildcane grass fibers is lower than for established fibers like sisal, coir, and banana, which is an attractive parameter in manufacturing light weight material. The diameter of different wildcane grass fibers under consideration varies between 190 and 560 μm. The yield of these fibers extracted by the retting process was 64% whereas with that of chemical process was only 35% by weight. This may be due to the removal of lignin to the maximum extent by chemical extraction. Even though the tensile strength of wildcane grass fiber is better than that of coir fiber only, the tensile modulus is more than those of sisal and banana fibers and much higher than that of coir fiber. The percentage elongation at break is also much less than those of coir, sisal, and banana fibers.

A typical load–deflection curve for highest volume fraction of all wildcane grass fiber composites are presented in Fig. 2. It is observed that all the curves show a linearly increasing trend up to a certain value of load and suddenly drops due to failure of specimens, and the arrest

Table 2 Comparison of the tensile properties of wildcane grass fibers along with other natural fibers [36–38]

Fiber name	Density (kg/m^3)	Diameter (μm)	Tensile strength (MPa)	Tensile modulus (GPa)	% elongation at break
Sisal	1450	80–300	227–700	9–20	3–14
Coir	1150	100–460	131–175	2.5–6	15–40
Banana	1350	80–250	529–759	8–20	1–3.5
Wildcane grass	844	190–560	159	11.84	1.34

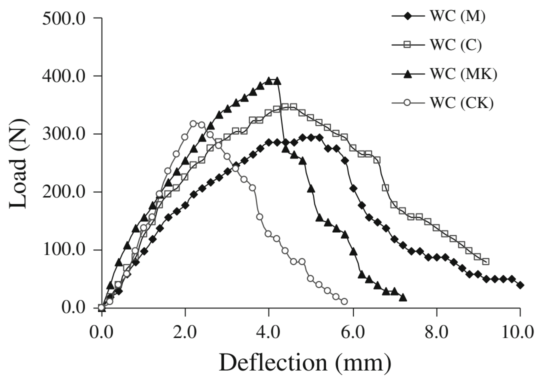

Fig. 2 Load against deflection characteristic at higher volume fraction for various wildcane grass fiber composites

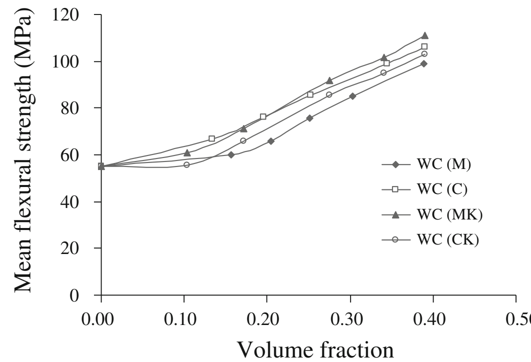

Fig. 3 Effect of volume fraction of fiber on mean flexural strength of various wildcane grass fiber composites

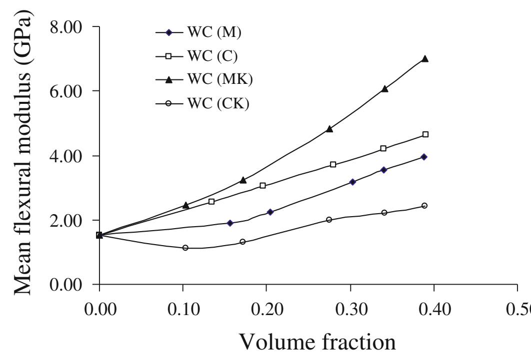

Fig. 4 Effect of volume fraction of fiber on mean flexural modulus of various wildcane grass fiber composites

points correspond to breakage and pull out of individual fibers from the resin matrix. The flexural resistance shown by various wildcane grass fiber composites increases in the order of wildcane grass (M), wildcane grass (CK), wildcane grass (C), and wildcane grass (MK). This is due to the improved adhesion between the resin and the fiber. The flexural modulus is calculated from the initial close to linear portion of the load–deflection curve and the maximum composite stress at the point of maximum load.

Ultimate flexural strength and modulus of various wildcane grass fiber-reinforced composites are presented in Table 3 for comparison. The effect of volume fraction of various wildcane grass fibers on flexural strength and modulus of composites is shown in Figs. 3 and 4. It is observed that as the volume fraction of fiber increases in the composite, the flexural strength of all fiber-reinforced composites increases in the order of wildcane grass (M), wildcane grass (CK), wildcane grass (C), and wildcane grass (MK). As the volume fraction of fiber increases beyond 0.20, it is observed that the flexural strength of wildcane grass (MK) fiber composite dominates that of wildcane grass (C) fiber composite. At the maximum volume fraction of 0.39, the flexural strength of wildcane grass (C) fiber composites have increased by approximately 7% to those of composites made from fibers extracted by retting process. The flexural strength of wildcane grass (MK) fiber composites have increased by 12% whereas that of

Table 3 The flexural properties of various wildcane grass fiber-reinforced composites at highest volume fraction of fiber (Standard deviation is given in brackets)

Composite	Volume fraction of fiber (V_f)	Flexural strength (MPa)	Flexural modulus (GPa)
Plain polyester	0.0	55.08 (±3.42)	1.535 (±0.16)
Wildcane Grass (M)	0.39	99.17 (±5.42)	3.96 (±0.17)
Wildcane Grass (C)	0.39	106.2 (±8.03)	4.63 (±0.51)
Wildcane Grass(MK)	0.388	111.0 (±6.45)	7.0 (±1.3)
Wildcane Grass (CK)	0.391	103.2 (±9.64)	2.43 (±0.23)

wildcane grass (CK) fiber composites have decreased by 3% compared to those of composites made from fibers extracted by retting and chemical processes, respectively. In spite of surface treatment of fiber, the flexural strength of wildcane grass (CK) fiber composites have decreased, which may be due to cell wall thickening that leads to poor adhesion with polyester resin as observed by earlier investigators [21].

As the volume fraction of fiber increases in the composite, the flexural modulus of different wildcane grass fiber composites increases in the order of wildcane grass fiber (CK), wildcane grass (M), wildcane grass (C), and wildcane grass (MK). At the maximum volume fraction of 0.39, the flexural modulus of wildcane grass (MK) fiber composite has increased by 76% over wildcane grass (M) fiber composite. This enhancement is attributed to the improved wetting of treated fiber with the polyester matrix. The flexural modulus of wildcane grass (CK) fiber composites has decreased by 48% compared to that of wildcane grass (C) fiber composites at the same volume fraction of fiber. The effect of volume fraction of fiber in the composite on the specific values of flexural strength and modulus are also shown in Figs. 5 and 6. As the volume fraction of fiber increases, the specific flexural strength of wildcane grass (MK) fiber and wildcane grass (CK) fiber composites exhibited nearly same value from 0.22 volume fraction of fiber to maximum volume fraction of fiber 0.39. This is due to higher composite density of wildcane grass (MK) fiber composite compared to that of wildcane grass (CK) fiber composite and wildcane grass fiber (MK) composite exhibits highest specific flexural modulus compared to all other composites considered in this study.

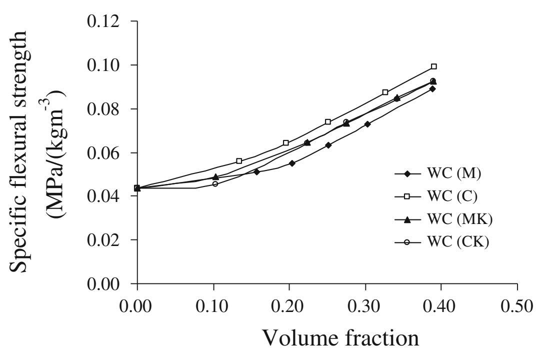

Fig. 5 Effect of volume fraction of fiber on specific flexural strength of various wildcane grass fiber composites

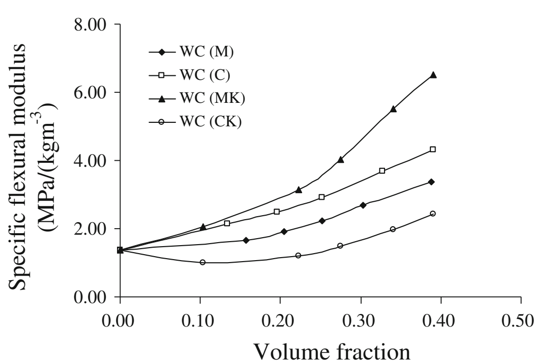

Fig. 6 Effect of volume fraction of fiber on specific flexural modulus of various wildcane grass fiber composites

Several researchers have reported that the flexural strength and modulus of polyester composites made by reinforcing different natural fibers like sisal, jute, coir, hemp, and kenaf treated by various methods have increased considerably compared to those of untreated fiber composites (Table 1). In this study, the flexural strength and modulus of wildcane grass (MK) fiber composite have increased by 12 and 76%, respectively, over those of wildcane grass (M) composite and the flexural modulus is also greater than those of sisal and jute fiber-reinforced polyester composites treated by different methods. However, the flexural properties of various wildcane grass fiber composites considered in this study are in good agreement to the composites made from other natural fibers as presented by earlier investigators.

Conclusions

The tensile modulus of wildcane grass fiber is relatively more than those of sisal and banana fibers and much higher than that of coir fiber. The flexural strength and modulus of all fiber-reinforced composites considered in this study increases with increase in fiber loading. The flexural strength and modulus of wildcane grass fiber (MK) composite is higher than those of other fiber-reinforced composites and wildcane grass fiber (MK) composite also exhibits highest specific flexural modulus compared to other composites considered in this study.

Wildcane grass is an abundantly available natural resource, which is renewable, and the extraction of its fiber is simple and economical. And also, low density of wildcane grass fiber compared to established fibers like sisal, banana, and bamboo, and high modulus makes an attractive parameter in manufacturing light weight material with high toughness.

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